SEP 2 5 2003

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of	Irene Spitsberg	:	
		:	
Serial No.	09/900,546	:	Group Art Unit 1762
		:	
Application Filed	July 6, 2001	:	Examiner Timothy Howard Meeks

For: METHOD FOR IMPROVING THE TBC LIFE OF A SINGLE PHASE PLATINUM ALUMINIDE BOND COAT

AFFIDAVIT UNDER 37 CFR § 1.131

Irene T. Spitsberg, being duly sworn, deposes and says:

1. I am the sole inventor of all the claims of the patent application identified above and am the inventor of the subject matter described and claimed therein.

2. Prior to February 22, 2000, I had completed my invention as described and claimed in the subject application in this country as evidenced by the following:

    a. Prior to February 22, 2000, having earlier conceived the idea of a method for coating a turbine blade for use in an aircraft engine with a thermal barrier coating system using a controlled preoxidation heat treatment which forms a thin layer of pure alumina over a single phase platinum aluminide, the thin layer of pure alumina being formed by a partial pressure of oxygen.

    b. Prior to February 22, 2000, an invention disclosure was provided to the GE Aircraft Engine Patent Department, a copy of which is attached. This disclosure sets forth the invention described in my patent application.

    c. After providing the invention disclosure to the GE Aircraft Engine Patent Department, but prior to February 22, 2000, I issued confidential internal GE Technical Memoranda entitled "Effect of Prexodiation at Controlled Oxygen Partial Pressure on TBC Spallation Life (Part I)" and "Effect of Preoxidation at Controlled Oxygen Partial Prssure on TBC Spallation Life (Part II); Evaluation of Oxide Films Grown on PtAl Surfaces under Different Partial Pressures of Oxygen", copies of relevant portions of which are attached.

    d. At some time after my submittal of the invention disclosure to the GE Aircraft Engine Patent Department and before March 30, 2000, a GE Patent Review Board determined that my invention disclosure should be filed as a patent application.

    e. On March 30, 2000, the invention disclosure was received by our outside patent attorney, with instructions to file a patent application.

    f. On April 6, 2000, a search was commissioned from a search firm.

g. On or about April 24, 2000, the search was returned by the search firm to our outside patent attorney.

h. Between May 1, 2000 and the filing date of the patent application, our outside patent attorney evaluated the search report and provided several drafts of the patent application, to which I provided comments which were subsequently incorporated.

i. On June 20, 2001, a final version of the patent application was forwarded by the outside patent attorney to in-house GE Patent Counsel.

j. The application was filed with the U.S. Patent and Trademark Office on July, 6, 2001.

3. Each of the dates deleted from the attached Exhibits is prior to February 22, 2000.
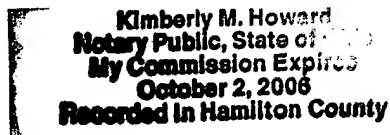

Irene Spitsberg

Irene Spitsberg

Sworn to and subscribed before me
this _17th_ day of September, 2003

Kimberly M. Howard

Notary Public

My Commission expires *October 2, 2006*

Kimberly M. Howard
Notary Public, State of
My Commission Expires
October 2, 2006
Recorded In Hamilton County

Exhibits

Part 1

Table of content.

Effect of preoxidation at controlled oxygen partial pressure on TBC spallation life
(Part I)

Abstract summary

This work was another attempt to develop a new approach to the PtAl bond coat preoxidation. Basis for this approach·has been developed through the following steps:
- revising the previous GEAE experience on the bond·coat preoxidation
- analyzing currently developed and published in literature knowledge on growth and properties of the aluminum oxide
- growing oxide films on the surface of PtAl bond coat at some selected conditions and characterizing their chemistry and microstructure (reported in Part II of this report)

Based on this analysis, a new set of experimental conditions for the bond coat preoxidation has been proposed and validated in the FCT test.

. Major finding were the following:

- Up to 1.5x life improvement is feasible in conventional vacuum. argon, hydrogen furnaces, whereas 2x + FCT life improvement possible in controlled oxygen atmosphere at pressures of oxygen <u>higher then 10^{-5} Torr.</u> Variation in FCT life is reduced as compared with preoxidation in air.

- Preoxidation outside coater preheat chamber might be sufficient since no difference in TBC performance was seen when preoxidation was done in the EB-PVD coater preheat chamber with the smooth transition into the coater for the TBC application as compared with the preoxidation in vacuum and hydrogen furnaces with cool down and a time period allowed before the TBC was applied

- Preoxidation in controlled oxygen atmosphere results in change in the TBC failure mode. lower rate of the oxide growth and no defects developed at the oxide/bond coat interface by the time of failure as seen with the resolution of SEM. This suggests that the preoxidation in low oxygen pressures resulted in denser and tougher oxide, and improved oxide adhesion.

Acknowledgments

Introduction

Oxidation of Ni-Al type alloys was studied by many people over last 10 years. It is generally known that dense and uniform film of α alumina oxide acts as oxidation resistance barrier due to low rate of oxygen diffusion through α alumina. Forming of such a film on the alloy/bond coat (in case of environmental and TBC coatings) results in slow rate of the oxide growth and, thus, longer time before the spallation of the oxide driven by CTE mismatch between the oxide and metal occurs. It is well known that properties of ceramic/bond coat interface determine spallation resistance of the TBC system. Properties of alumina layer growing between the ceramic topcoat and the bond coat as well as the rate of the layer growth have the major impact on the stresses generated at the critical interface and the interface strength. So, the idea itself that the bond coat preoxidation treatment can result in "a better" properties of the grown oxide and/or reduce the oxide growth rate (by altering the oxide microstructure and phase composition) has been developed in many papers. However, it is still not clear what exactly properties or their combination of the oxide film should be achieved to benefit the TBC spallation life

The approach of preoxidation of PtAl bond coat to improve TBC FCT life was tried at GEAE over last 5-6 years by Jon Schaeffer, Scott Walston, Robert Bruce, Dave Wortman and Tony Maricocchi. Even though some significant TBC life improvement was demonstrated in many cases, which proved feasibility of the approach of the bond coat preoxidation, it seemed impossible to get stable and reproducible results. (This experience is fully documented in Scott Walston's report, TM 99-4, and referred to in greater details in the text of this report). It was decided in 1996 to discontinue the preoxidation work till better understanding of the aluminum oxide growth and it's implications are developed.

This work was another attempt to develop a new approach to the PtAl bond coat preoxidation. Basis for this approach has been developed through the following steps:
* revising the previous GEAE experience on the bond coat preoxidation
* analyzing currently developed and published in literature knowledge on growth and properties of the aluminum oxide
* growing oxide films on the surface of PtAl bond coat at some selected conditions and characterizing their chemistry and microstructure

Based on this analysis, a new set of experimental conditions for the bond coat preoxidation has been proposed and validated in the FCT test.

1. Literature search on oxidation of NiAl type alloys.

1.1 Effect of alloy (/bond coat) chemistry on chemical composition of the oxide scale. General Effects of temperature and oxygen pressure.

7. No contaminates at the oxide/bond coat interface after the oxide formation was observed at any surface treatment.
8. 2 phase coating is prone to internal oxidation in low oxygen partial pressures due to selective oxidation of the phase boundaries. This suggests that optimal conditions for formation of thin dense aloha alumna oxide will be different for single and two phase coating.

The following recommendations could have been made:

Phase composition of the oxides has to be further determined with XRD and TEM techniques. Nevertheless, *it seems reasonable to assume, based on the reported observations ,that it is likely that alpha alumina oxide can be formed at 2050F within 1 to 3 hr. Microstructure of the oxide, however, depends, on the oxidation atmosphere and surface conditions. It seems that formation of comact alpha alumina film is possible at oxygen content higher the 10^{-5} Tor but with the oxygen partial pressure still low, since it is seen that in air not uniform and cracked oxide can easily be formed. It also seems that surface techniques like grit blasting, which removes the surface layer without selective attack on the coating major components, should be used. Based on the scale of the oxide grains and observed process of the oxide growth, it is not thought that a certain roughness is what matters, provided that some fine roughness created by a mechanical treatment.*

Based on this, the specimens identical to the specimens exposed at low oxygen pressures were coated with TBC and FCT tested at 2125F. The data on preoxidation in air for single phase PtAl at 2050F were taken form the Scott Walston's previous work for the comparison.

5. Results.

Data in Table 1 and Figures 12-14 show effect of preoxidation in partial oxygen pressure on TBC FCT life at 2125F. Majority of the tested samples were MTL1000 VPA single phase PtAl on René N5 (MQTD VPA runs 990515 and 990517) with the standard grit surface treatment with 80 alumina grit/60 psi. The baseline FCT TBC life on the PtAl from these runs was 240-260 cycles at 2125F which is within the statistically expected range (240 +/- 40 cycles) for this coating type. Historical baseline data and data for preoxidation in air (by Scott Walston) are given in the Figures for the comparison.

Some samples with acid treated surface were tested as well.

The data suggest that:

- up to 1.5x life improvement feasible in conventional vacuum, argon, hydrogen furnaces

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- 2x + FCT life improvement possible in controlled oxygen atmosphere
- data suggest that preoxidation outside coater preheat chamber might be sufficient since no difference in TBC performance was seen when preoxidation was done in the EB-PVD coater preheat chamber with the smooth transition into the coater for the TBC application as compared with the preoxidation in vacuum and hydrogen furnaces with cool down and a time period allowed before the TBC was applied
- average FCT life in all outside the coater preoxidation vacuum runs is 318 +/- 26 cycles
- further improvement is possible with control of oxygen atmosphere. 420-600 cycles FCT life at 2125F was achieved when preoxidation was done at 10^{-4} Torr partial pressure of oxygen. (This was achieved by feeding a small amount of oxygen into the EB-PVD coater preheat chamber and controlling it at the designed level by OGC).
- PtAl coatings with acid treated surface did not show any life improvement over baseline samples under conditions where the improvement was seen with the grit blasted surface. This is thought to be due to the surface depletion with Al as a result of the acid treatment, as shown in part II of this report. Typical 2125F FCT life on the acid treated samples with no preoxidation treatment was around 220 cycles, whereas results between 180 and 280 cycles were observed with different preoxidation treatments.

Failure analysis was performed on the failed FCT samples. It was clearly seen that the preoxidation treatment at low oxygen pressures resulted in change of the TBC failure mode. The failure occurred for 100% at the oxide/TBC interface. It was clearly seen that the oxide convolutions occurred at the coating grain boundaries. However, instead of the oxide breakage, the TBC breakage was observed. This seems to initiate the crack which further propagated along the TBC.oxide interface. Surface of the failed samples showed intact oxide scale, with the grain boundary network being decorated by "broken" TBC. This suggests that toughness of the oxide formed as result of the preoxidation treatment was improved. It also could be noticed that thickness of the oxide grown for 420 cycles on the preoxidized in oxygen coating is about 7 micron that is typical oxide thickness observed on the baseline failed specimens (200 – 280 cycles). This suggests that the oxide growth rate is reduced by the preoxidation treatment. Another important observation is that the bond coat/oxide interface on the preoxidized specimens looks "defect free" whereas the baseline specimens show vacancies coagulation and fine pore formation at this interface at failure. These observations are summarized in Figure 15. and can be seen in much greater details on SEM micrographs in Appendix 1.
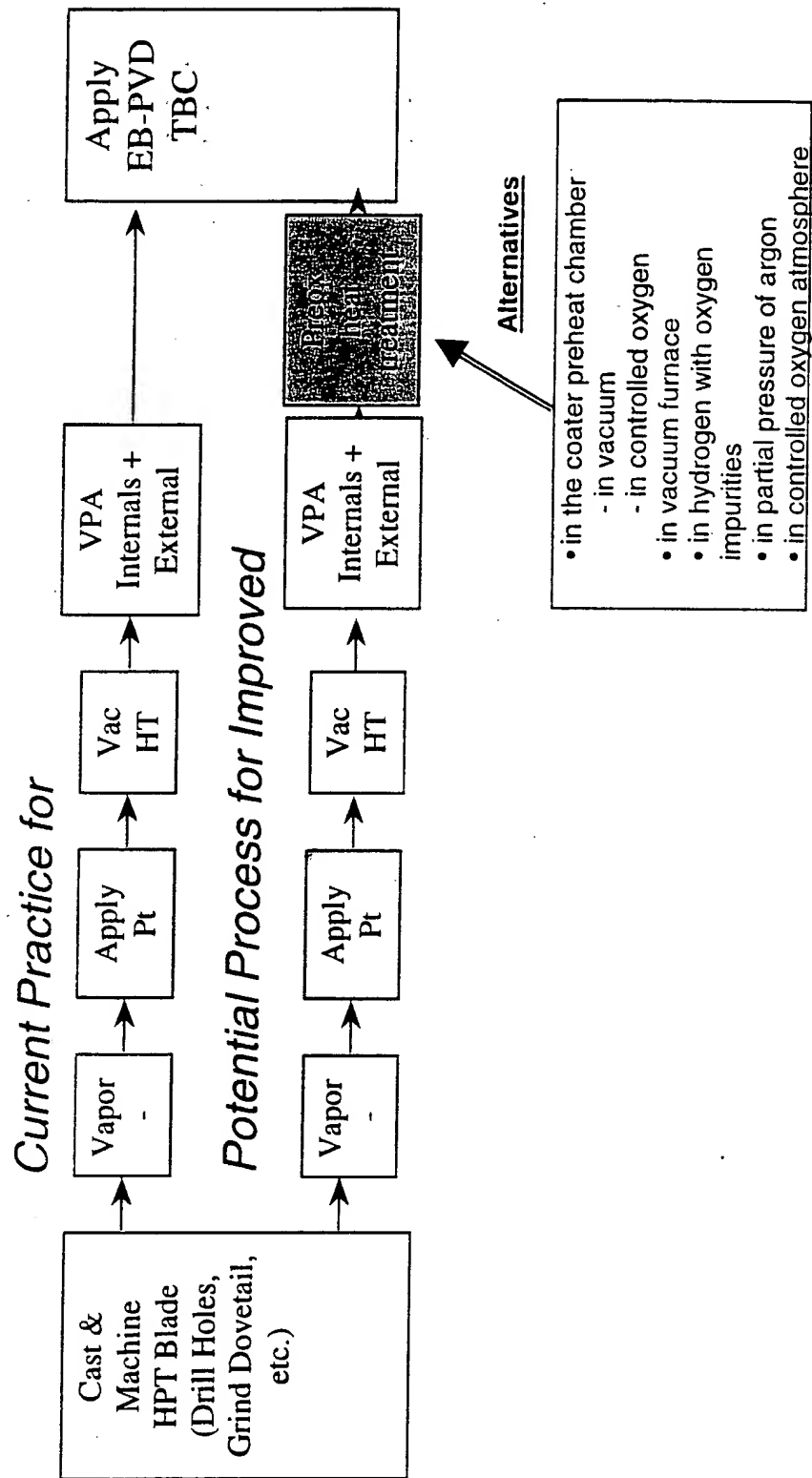
Process Flow for Improved PtAl by preoxidation

Figure 12.

Table 1. Statistical data on FCT life of preoxidized PtAl bond coats.

Descriptive Statistics

Variable FCT life Preoxida	N	Mean	Median	TrMean	StDev	
1	2	310.0	310.0	310.0	14.1	vac,2hr, coater
2	3	306.7	300.0	306.7	30.6	vac,4hr, coater
3	3	346.67	340.00	346.67	11.55	vac,4hr, coater, Howmet
4	6	306.67	320.00	306.67	24.22	vac,2hr, braze lab
5	6	306.67	300.00	306.67	10.33	Argon, 2 hr, braze lab
6	3	353.3	360.0	353.3	30.6	hydrogen, 4hr
7	3	520.0	520.0	520.0	100.0	Oxygen,2hr, coater
8	3	393.3	400.0	393.3	90.2	oxygen,4hr, Howmet

Variable FCT life Preoxida	SE Mean	Minimum	Maximum	Q1	Q3
1	10.0	300.0	320.0	*	*
2	17.6	280.0	340.0	280.0	340.0
3	6.67	340.00	360.00	340.00	360.00
4	9.89	260.00	320.00	290.00	320.00
5	4.22	300.00	320.00	300.00	320.00
6	17.6	320.0	380.0	320.0	380.0
7	57.7	420.0	620.0	420.0	620.0
8	52.1	300.0	480.0	300.0	480.0

Descriptive Statistics for preoxidation runs in 10 to -5 oxygen partial pressure

Variable	N	Mean	Median	TrMean	StDev	SE Mean
FCT life	23	318.26	320.00	318.10	26.91	5.61

Variable	Minimum	Maximum	Q1	Q3
C18	260.00	380.00	300.00	340.00

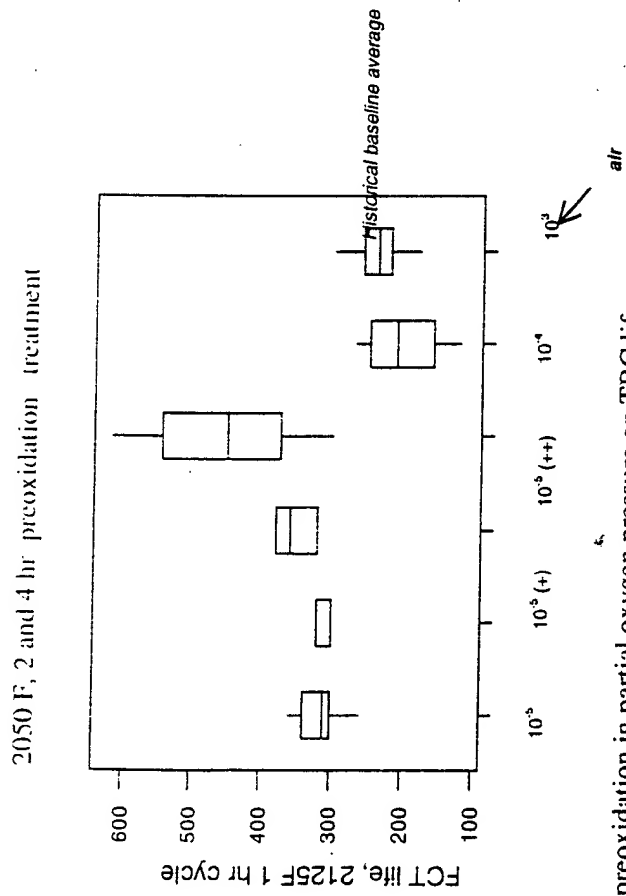2050 F, 2 and 4 hr preoxidation treatment

Figure 12. Effect of preoxidation in partial oxygen pressure on TBC life.

Figure 13. TBC FCT life in different preoxidation runs. (2125F 1 hr cycles). Preoxidation at 2050F for 2 and 4 hours.
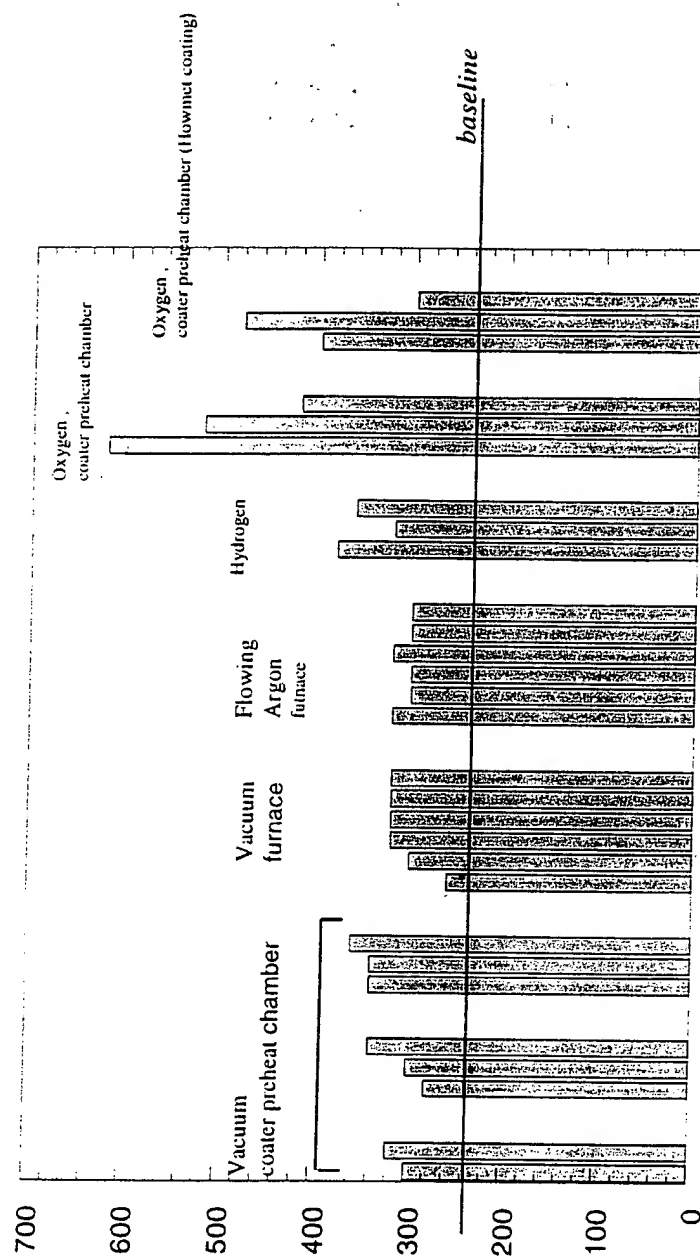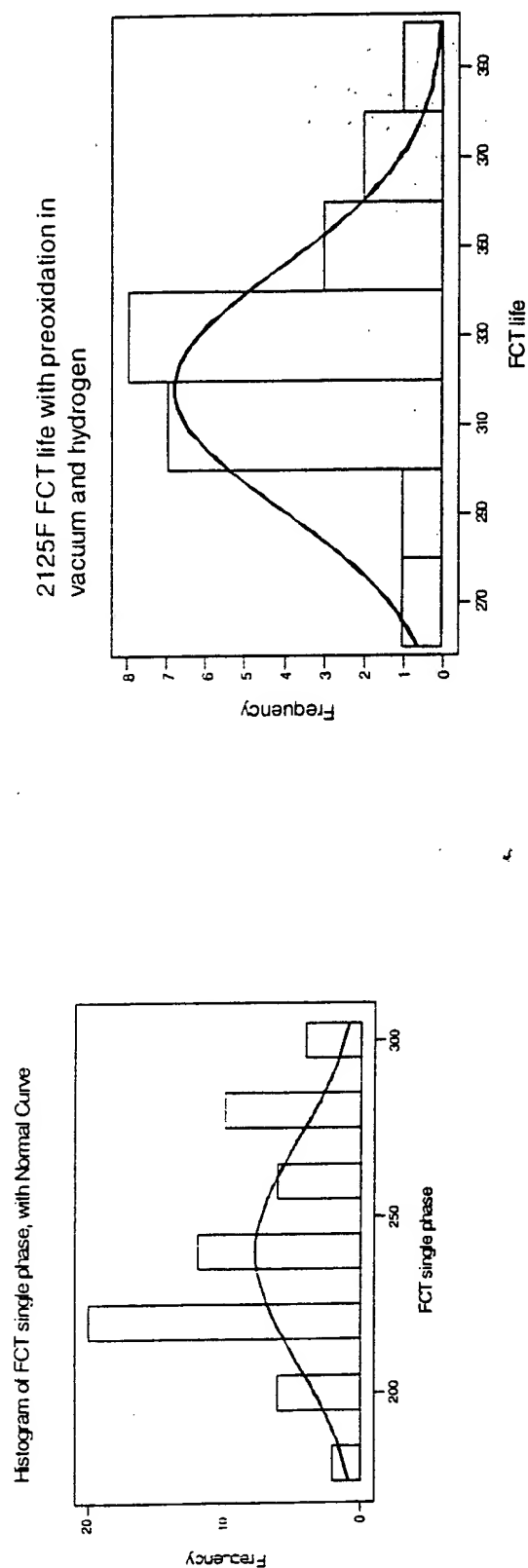
Figure 14. Statistical data showing effect of preoxidation in vacuum and hydrogen on TBC FCT life
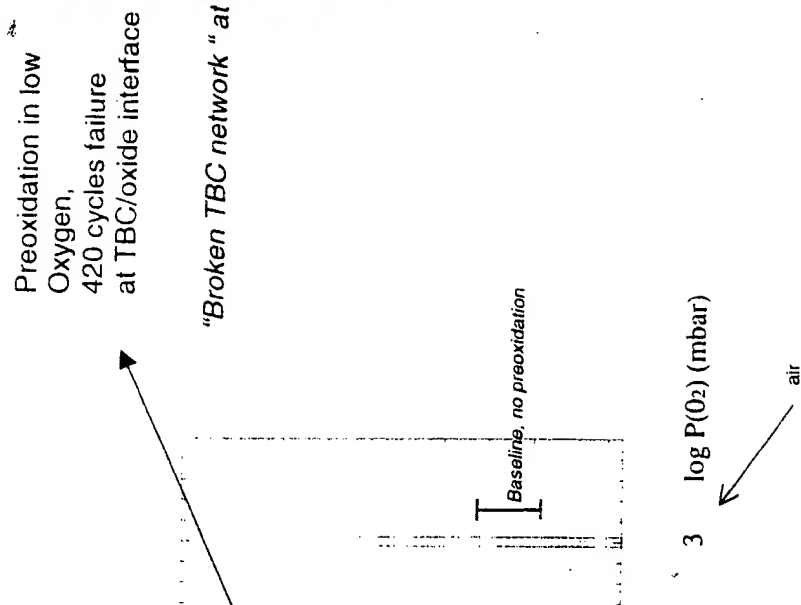
2125F FCT life with preoxidation in vacuum and hydrogen

Descriptive Statistics

Variable	N	Mean	Median	Tr Mean	StDev	SE Mean
FCT life	23	318.26	320.00	318.10	26.91	5.61

Variable	Min	Max	Q1	Q3
FCT life	260.00	380.00	300.00	340.00

Histogram of FCT single phase, with Normal Curve

escriptive Statistics

ariable	N	Mean	Median	TrMean	StDev	SE Mean
CT sing	0	240.00	240.00	239.63	31.25	4.03

ariable	Minimum	Maximum	Q1	Q3
CT sing	180.00	300.00	220.00	260.00

Figure 15. Effect of Pre-Oxidation at Different Oxygen Partial Pressures on FCT life (N5/PtAl)

Preoxidation in low
Oxygen,
420 cycles failure
at TBC/oxide interface

"Broken TBC network " at coating

Baseline, no preoxidation

log P(O$_2$) (mbar)

air

700
600
500
400
300
200
100

-6   -5   -4   3

Baseline , oxide at failure
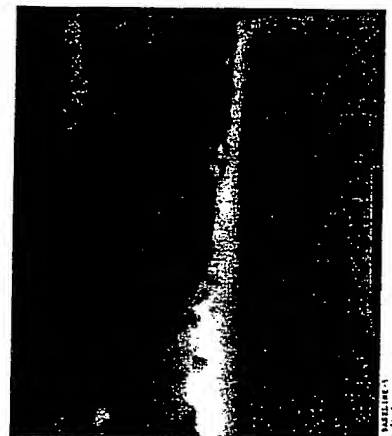
- **Oxygen control is important for**
  - **preoxidation**
- **Preoxidation changes TBC**
  - **failure mode**
- **Improved oxide/bond coat**
  - **interface with preoxidation**
  - **in low Oxygen**
- **Promising for oxidation resistance**
  - **improvement**
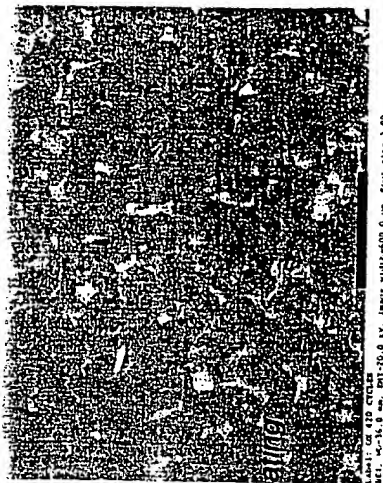
Conclusions

- Up to 1.5x life improvement feasible in conventional vacuum. argon. hydrogen furnaces

- 2x + FCT life improvement possible in controlled oxygen atmosphere at pressures of oxygen <u>higher then 10^{-5} Torr.</u>

- Data suggest that preoxidation outside coater preheat chamber might be sufficient since no difference in TBC performance was seen when preoxidation was done in the EB-PVD coater preheat chamber with the smooth transition into the coater for the TBC application as compared with the preoxidation in vacuum and hydrogen furnaces with cool down and a time period allowed before the TBC was applied

- Average FCT life in all outside the coater preoxidation runs is 318 +/- 26 cycles. with further improvement is possible with control of oxygen atmosphere

- 420-600 cycles FCT life at 2125F was achieved when preoxidation was done at 10^{-4} Torr partial pressure of oxygen. This was achieved by feeding a small amount of oxygen into the EB-PVD coater preheat chamber and controlling it at the designed level by OGC. It can be assumed that mixing small amount of oxygen into argon and caring the preoxidation treatment in a conventional argon furnace could provide TBC life benefit comparable with the one demonstrated in the coater preheat chamber in the oxygen containing atmosphere.

- Variation in FCT life is reduced as compared with preoxidation in air.

- Preoxidation in controlled oxygen atmosphere result in change in the TBC failure mode. lower rate of the oxide growth and no defects developed at the oxide/bond coat interface by the time of failure as seen with the resolution of SEM. This suggests that the preoxidation in low oxygen pressures resulted in denser and tougher oxide and improved oxide adhesion.

- It is proposed that the best performing oxides were characterized with TEM and the stress measuring techniques

- The next logical step would be to evaluate possibility of oxygen impurity level control through using argon/oxygen mixtures in a conventional vacuum/argon furnace.

References

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13. T.E. Stragman, P.A. Solfest, U.S. Patent 4, 880,614, "Ceramic Thermal Barrier Coatings with Alumina Barrier Interlayer".

14. W. Lih, E. Chang, B.C. Wau and C.H. Chao, Oxidation of Metals. Vol. 36, Nos ¾, 1991, pp. 221-138, " Effect of preoxidation on the properties of ZrO2-8wt.% Y2O3/Ni- 22Cr – 10Al – 1Y Thermal Barrier Coatings

Table of content.

Effect of preoxidation at controlled oxygen partial pressure on TBC spallation life (Part II):
Evaluation of oxide films grown on PtAl surface under different partial pressures of oxygen.

Abstract summary

The purpose of this experimental work was to evaluate microstructure and chemical composition of the oxides grown on surface of VPA PtAl coating (on Rene N5) at 2050 F in partial pressure of oxygen , from 10^{-5} Torr to 0.2 atm (air).

Three different surface finishes were evaluated: vapor hone at 1200 grit/60 psi with alumina grit , grit blasting at 80 grit/60 psi (with alumina grit at RDC); and acid etching with Aqua Regia (2 parts of HNO_3 : 3parts of HCl). Surface chemical analysis data is presented.

<u>Major findings were the following:</u>

1. Oxidation in air results in non-uniform oxide formation across the coating surface, with the oxide experiencing cracking upon cooling. Chemical composition of the oxide varies across the surface of the coating, with Ni and Cr being present in the oxide thickness.

2. Oxidation in low oxygen pressure typically results in uniform, compact oxide structure, with pure alumina being the predominate constituent. Minor Ni and Cr is typically observed in the top oxide layer (on nanometer scale).

3. It is possible that at short time of oxidation (1 hr and less) either no continuous film is formed yet, or the ridges from α to θ phase transformation might remain not completely healed. It seems that adding small amount of oxygen into vacuum atmosphere accelerates formation of the continuous oxide film with the most uniform dense microstructure.

4. In many cases the "ridge" oxide structure typical of alpha alumina oxide was observed at oxidation in low oxygen partial pressure. It was always true when oxidation was done with added oxygen. In vacuum, however, "blade" type of oxide structure characteristic of theta alumina oxide was observed after 1 hr of oxidation

5. Grit blasting of the surface promotes formation of uniform oxide with the "ridge" type structure of alpha alumina.

6. Acid surface treatment leaches out the aluminum and, thus retards formation of continuous film in some cases whereas might promote transitional oxides formation in other cases (oxidation in air).

7. No contaminates at the oxide/bond coat interface after the oxide formation was observed at any surface treatment.

8. 2 phase coating is prone to internal oxidation in low oxygen partial pressures due to selective oxidation of the phase boundaries. This suggests that the optimal conditions for formation of thin, dense alpha alumna oxide will be different for single and two phase coating.

The following recommendations made:

Phase composition of the oxides has to be further determined with XRD and TEM techniques. Nevertheless, *it seems reasonable to assume, based on the reported observations, that it is likely that alpha alumina oxide can be formed s at 2050F within 1 to 3 hr. Microstructure of the oxide, however, depends on the oxidation atmosphere and surface conditions. It seems that formation of compact alpha alumina film is possible at oxygen content higher the 10^{-5} Torr but with the oxygen partial pressure still low, since it is seen that in air not uniform and cracked oxide can easily be formed. It also seems that surface techniques like grit blasting, which removes the surface layer without selective attack on the coating major components, should be used. Based on the scale of the observed oxide grains and process of the oxide growth, it is not thought that a certain roughness is what matters, provided that some fine roughness created by a mechanical treatment.*

Next logical step would be to validate these proposed trends in FCT test. This work is in progress and partially reported in Part I of this report " Effect of oxygen partial pressure in preoxidation treatment of PtAl bond coat on TBC life".

Acknowledgments

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usage has long been
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n and reduction al-
:dox reactions), and
ns is termed the ox-
ipric ion is the oxi-
iction: Fe (metal) +
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by loss of two elec-
:s the two electrons
ed). Electrons may
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Such partial loss of
oxidation in its
application of the
ses that at first sight
ions. Reaction of a
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double bond is re-

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: A substance
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that of the reduced form and can be reversibly oxidized and reduced. Thus, if diphenylamine is present in a ferrous sulfate solution to which potassium dichromate is being added, a violet color appears with the first drop of excess dichromate.
See indicator.

oxidative coupling. A polymerization technique for certain types of linear high polymers. Oxidation of 2,6-dimethylphenol with an amine complex of a copper salt as catalyst forms a polyether, with splitting off of water. The product is soluble in aromatic and chlorinated hydrocarbons; insoluble in alcohols, ketones, and aliphatics. It is thermoplastic and unaffected by acids, bases, and detergents. It has a very broad useful temperature range (from −170 to +190C). It is also dimensionally stable and has good electrical resistance. Oxidative coupling of diacetylenes and dithiols also yields promising polymers.
See "PPO."

oxide. A mineral in which metallic atoms are bonded to oxygen atoms.

oxidizing material. Any compound that spontaneously evolves oxygen either at room temperature or under slight heating. The term includes such chemicals as peroxides, chlorates, perchlorates, nitrates, and permanganates. These can react vigorously at ambient temperatures when stored near or in contact with reducing materials such as cellulosic and other organic compounds. Storage areas should be well ventilated and kept as cool as possible.

oxine. See 8-hydroxyquinoline.

oxirane.
CAS: 75-21-8.

H₂COCH.
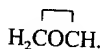
A synonym for ethylene oxide. An oxirane group is one having the structure

=COC=

and is one kind of epoxy group.
See ethylene oxide.

Oxirane process. A method of making ethylene glycol by catalytic oxidation of ethylene to the diacetate, which is then hydrolyzed to ethylene glycol.

oxirene. (oxacyclopropene). An organic intermediate containing four πM electrons, reported to result from oxidation of acetylene.

2-oxohexamethylenimine. See caprolactam.

"Oxone" [Du Pont]. TM for an acidic, white, granular, free-flowing solid containing the active ingredient potassium peroxymonosulfate; readily soluble in water; 1% solution has pH of 2–3; minimum active oxygen content 4.5%; strong oxidizing agent.
Hazard: Fire risk in contact with organic materials.
Use: Manufacture of dry laundry bleaches, detergent-bleach washing compound, scouring powders, plastic dishware cleaners, and metal cleaners; hairwave neutralizers, pharmaceuticals; general oxidizing reactions.

oxonium ion. See hydronium ion.

2-oxopentanedioic acid. See α-ketoglutaric acid.

4-oxopentanoic acid. See levulinic acid.

Oxo process. Production of alcohols, aldehydes, and other oxygenated organic compounds by passage of olefin hydrocarbon vapors over cobalt catalysts in the presence of carbon monoxide and hydrogen. Aldehydes are formed as products, but in most cases these are hydrogenated at once to the corresponding alcohol. Propylene produces normal and isobutyraldehyde; higher olefins produce a mixture of aldehydes containing one more carbon atom than the olefins; *n*-butyl, isobutyl, amyl, isooctyl, decyl, and tridecyl alcohols are produced in large quantities.

oxosilane. See siloxane.

oxybenzoic acid. See hydroxybenzoic acid.

oxybenzone. (4-methoxy-2-hydroxybenzophenone).
CAS: 131-57-7. C₁₄H₁₂O₃.
Properties: Colorless crystals. Mp 65C. Soluble in common organic solvents.
Use: Sunscreen lotions.

***p,p'*-oxybis(benzenesulfonylhydrazide).**
[(4,4'-oxybis(benzenesulfonyl)hydrazide)].
H₂NNHSO₂C₆H₄OC₆H₄SO₂NHNH₂.
Properties: Fine, white, crystalline powder; odorless. D 1.52, mp decomposes at 150–160C. Soluble in acetone; moderately soluble in ethanol and polyethylene glycols; insoluble in gasoline and water. Combustible.
Use: Blowing agent for sponge rubber and expanded plastics.

oxyconiine. See conhydrine.

oxydemetonmethyl. (generic for *S*-[2-(ethylsulfinyl)ethyl]-*O,O*-dimethylphosphorothioate).
See *O,O*-dimethyl-*S*-2-(ethylsulfinyl)ethyl phosphorothioate.

***n*-oxydiethylene-2-benzothiazolesulfenamide.**
See 2-(morpholinothio)benzothiazole.